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(21) International Application Number: PCT/EP97/05762 (22) International Filing Date: 13 October 1997 (13.10.97) (30) Priority Data: 96307507.2 16 October 1996 (16.10.96) EP <i>(34) Countries for which the regional or international application was filed:</i> AT et al. (71) Applicant (for AU BB CA GB GH IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU BB CA GB GH IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG US ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (72) Inventors; and (75) Inventors/Applicants (for US only): GRAINGER, David, Stephen [GB/GB]; 27 Prescott Street, Hoole, Chester CH2 3BN (GB). KHAN-LODHI, Abid, Nadim [GB/US]; 6-1 Bergen Ridge Road, North Bergen, NJ 07047 (US).		(74) Agent: TANSLEY, Sally, Elizabeth; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: FABRIC SOFTENING COMPOSITION. (57) Abstract A fabric softening composition that softens fabrics without affecting the absorbency of the fabric is provided. The conditioner comprises: i) a liquid or soft solid derivative of a cyclic polyol (CEP) or of a reduced saccharide (RSE) resulting from 35 to 100 % of the hydroxyl groups in the cyclic polyol or reduced saccharide being esterified or etherified, the derivate (CPE or RSE) having at least 2 or more of ester or ether groups independently attached to a C ₈ -C ₂₂ alkyl or alkenyl chain or mixtures thereof, and containing at least 35 % tri or higher esters; ii) a deposition aid. Also liquid or soft solid fatty acid esters of glucose comprising a mixture of C ₂ -C ₂₂ alkyl or alkenyl chains and resulting from a cyclic polyol as above are provided.		

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FABRIC SOFTENING COMPOSITION5 Technical Field

The present invention relates to fabric softening compositions. In particular the invention relates to fabric softening compositions that soften fabrics without affecting
10 the absorbency of the fabric.

Background and Prior Art

15 Rinse added fabric softener compositions are well known. However, a disadvantage associated with conventional rinse conditioners is that although they increase the soft feel of a fabric they simultaneously decrease the fabric's absorbency. A decrease in the absorbency properties of a
20 fabric means that its ability to take up water decreases. This is particularly disadvantageous with towels where the consumer requires the towel to be soft, and yet, have a high absorbency.

25 Co-pending application GB 9518012.1 (Unilever) discloses liquid fabric conditioning compositions comprising a substantially water insoluble fabric softening compound. The compositions have a phase structure with greater than one-dimensional long range order as defined by x-ray
30 crystallography. The compositions are said to exhibit good softening and hydrophobicity.

EP 0 380 406 (Colgate-Palmolive) discloses detergent compositions comprising a saccharide or reduced saccharide ester containing at least one fatty acid chain.

5

Bleaching detergent compositions comprising derivatives of pentoses or hexoses, having a long chain acyl or aroyl group and one or more short chain alkyl groups attached through a glycosidic bond are disclosed in WO 91/10719 (Novo).

10

Textile treatment compositions containing mixtures of cationic and nonionic surfactants are disclosed in GB 1 601 359 (Procter & Gamble).

15

US 4 800 038 (Colgate-Palmolive) discloses the use of acylated sugar ethers as a bleach activator in nonionic detergent compositions.

20

WO 9500614 (Kao Corporation) discloses softening compositions comprising polyhydric alcohol esters and cationised cellulose.

25

JP 06264049 (Mitsubishi Kasei Corporation) discloses viscosity improving sucrose long chain fatty acid esters with an average degree of substitution of 4 to 7 in cosmetics and topical skin compositions.

30

US 5 447 643 (Hüls) discloses aqueous fabric softeners comprising nonionic surfactant and mono, di or tri fatty acid esters of certain polyols.

WO 96/15213 (Henkel) discloses textile softening agents containing alkyl, alkenyl and/or acyl group containing sugar derivatives, which are solid after esterification, in combination with nonionic and cationic emulsifiers.

5

The present invention is directed towards alleviating the problems associated with the prior art as referred to hereinabove.

10

The principal advantages of the present invention are that its' compositions when used to soften fabrics do so effectively without detriment to the absorbency of the fabric and that the compositions are easily manufactured.

15

Definition of the Invention

Thus according to one aspect of the invention there is provided:

20

A fabric softening composition comprising:

- 25 i) a liquid or soft solid derivative of a cyclic polyol (CPE) or of a reduced saccharide (RSE) resulting from 35 to 100% of the hydroxyl groups in the cyclic polyol or reduced saccharide being esterified or etherified, the derivative (CPE or RSE) having at least 2 or more of ester or ether groups independently attached to a C₈-C₂₂ alkyl or alkenyl chain or mixtures thereof, and containing at least 35% tri
30 or higher esters and;

ii) a deposition aid.

The invention also relates to a method of treating fabric comprising the step of treating the fabrics with a composition comprising a CPE or RSE wherein the CPE or RSE is a liquid or soft solid having a solid:liquid ratio between 50:50 and 0:100 at 20°C as determined by T₂ relaxation time NMR.

The invention also discloses the use of a CPE or RSE as a fabric softening aid that does not decrease the fabric's absorbency.

The invention further relates to a liquid or soft solid fatty acid ester of glucose having a solid:liquid ratio between 50:50 and 0:100 as measured above, the ester groups comprising a mixture of C₂-C₂₂ alkyl or alkenyl chains and resulting from a cyclic polyol having 35 to 100% of the hydroxyl groups esterified. The use of a sorbitan mono ,di or trioleate as a hydrophilic softener is also provided. It has been found, surprisingly, that the above compositions provide an unexpected combination of simultaneous fabric softening and retention of absorbency.

25

Detailed Description of the Invention

In the context of the present invention the initials CPE or RSE stand for a cyclic polyol derivative or a reduced saccharide derivative respectively which results from 35 to 100% of the hydroxyl groups of the cyclic polyol or reduced

saccharide being esterified and/or etherified and in which at least two or more of ester or ether groups are independently of one another attached to a C₈ to C₂₂ alkyl or alkenyl chain.

5

In the fabric softening compositions of the invention the ratio of CPE or RSE to deposition aid is preferably within the range of from 1:10 to 15:1, more preferably within the range from 1:5 to 10:1, most preferably 1:1 to 10:1.

10

If a composition is required to give particularly high absorbency to fabrics it is advantageous if the ratio of CPE or RSE to deposition aid is at least 1:1 (especially if the deposition aid is a fabric softening compound).

15

If a highly softening composition is required it is advantageous if the ratio of a softening deposition aid to CPE or RSE is at least 2:3, preferably 1:1.

20

To give excellent softening and hydrophobicity to fabrics it is preferred if the ratio of softening deposition aid to CPE or RSE is from 3:2 to 1:10, more preferably from 2:3 to 1:10.

25

The CPE or RSE

30

The liquid or soft solid (as hereinafter defined) CPEs or RSEs of the present invention result from 35 to 100% of the hydroxyl groups of the starting cyclic polyol or reduced saccharide being esterified or etherified. Typically the CPE's or RSE's have 3 or more ester or ether groups or

mixtures thereof for example 4 or more, example 5 or more.
It is preferred if two or more of the ester or ether groups
of the CPE or RSE are independently of one another attached
to a C₈ to C₂₂ alkyl or alkenyl chain. The C₈ to C₂₂ alkyl or
5 alkenyl groups may be branched or linear carbon chains.

Preferably 40 to 100% of the hydroxyl groups, most
preferably 50 to 100% are esterified or etherified.

10 CPEs are preferred for use with the present invention.
Inositol is a preferred example of a cyclic polyol.
Inositol derivatives are especially preferred.

In the context of the present invention the term cyclic
15 polyol encompasses all forms of saccharides. Indeed
saccharides are especially preferred for use with this
invention. Examples of preferred saccharides for the CPE's
or RSE's to be derived from are monosaccharides and
disaccharides.

20 Examples of monosaccharides include xylose, arabinose,
galactose, fructose, sorbose and glucose. Glucose is
especially preferred.

25 An example of a reduced saccharide is sorbitan.

Examples of disaccharides include maltose, lactose,
cellobiose and sucrose. Sucrose is especially preferred.

30 The liquid or soft solid CPE's or RSE's of the present
invention can be prepared by a variety of methods well known

to those skilled in the art. These methods include acylation of the cyclic polyol or reduced saccharide with an acid chloride; trans-esterification of the cyclic polyol or reduced saccharide fatty acid esters using a variety of catalysts; ; acylation of the cyclic polyol or reduced saccharide with an acid anhydride and acylation of the cyclic polyol or reduced saccharide with a fatty acid. Typical preparations of these materials are disclosed in US 4 386 213 (Procter and Gamble) and AU 14416/88 (Procter and Gamble).

It is preferred if the CPE or RSE has 4 or more ester or ether groups although for some compounds 3 ester or ether groups produce excellent results and are preferred. If the cyclic CPE is a disaccharide it is preferred if the disaccharide has 4 or more ester or ether groups. Particularly preferred CPE's are esters with a degree of esterification of 4 or more, for example, sucrose esters.

It is advantageous if the saccharide or reduced saccharide has 3 or more ester groups.

Where the cyclic polyol is a reducing sugar it is advantageous if each ring of the CPE has one ether group, preferably at the C₁ position, and the remaining hydroxyl groups are esterified. Suitable examples of such compounds include methyl glucose derivatives.

Examples of suitable CPEs include esters of alkyl(poly)glucosides, in particular alkyl glucoside esters having a degree of polymerisation from 1 to 2.

It is preferable if the ester or ether groups of the cyclic polyol or reduced saccharide are independently of one another attached to a C₈ to C₂₂ alkyl or alkenyl chain or a C₂ to C₈ chain and in which the ratio of C₈ to C₂₂ groups to C₂ to C₈ is from 5:3 to 3:5. This is because these products are in the main oils (liquids) and are thus easy to formulate. It has also been found that excellent results are achieved with oils (liquids).

Examples of suitable saccharide esters include fatty acid esters of glucose, the ester groups comprising C₂-C₁₈ alkyl or alkenyl chain and the degree of esterification being 5. In particular saccharides having ester groups consisting essentially of a C₂ alkyl chain and a C₈ to C₁₂ straight alkyl chain, the molar ratio of short chain C₂ alkyl chains to C₈ to C₁₂ straight alkyl chains being from 2:1 to 1:2, more preferably about 1:1 are suitable. It is further preferred if the degree of esterification is at least 5.

The liquid or soft solid CPE's or RSE's of the present invention are characterised as materials having a solid:liquid ratio of between 50:50 and 0:100 at 20°C as determined by T₂ relaxation time NMR, preferably between 43:57 and 0:100, most preferably between 40:60 and 0:100, such as, 20:80 and 0:100. The T₂ NMR relaxation time is commonly used for characterising solid:liquid ratios in soft solid products such as fats and margarines. For the purpose of the present invention, any component of the NMR signal with a T₂ of less than 100 μs is considered to be a solid

component and any component with $T_2 \geq 100 \mu s$ is considered to be a liquid component.

5 For the CPE's and RSE's the tetra, penta etc prefixes only indicate the average degrees of esterification. The compounds exist as a mixture of materials ranging from the monoester to the fully esterified ester. It is the average degree of esterification which is used herein to define the CPE's and RSE's.

10

It has been found that CPE's and RSE's having unsaturated or mixed alkyl chain lengths show advantageous results.

15

Factors governing the suitability of the CPE's and RSE's are the presence and degree of branched chains, mixed chain lengths and the level of unsaturation.

The Deposition aid

20

In the context of the present invention a deposition aid is defined as any material that aids deposition of the selected CPE or RSE onto a fabric during the laundering process.

25

The deposition aid may be selected from a fabric softening compounds, cationic compounds, nonionic surfactants, anionic surfactants, polymeric deposition aids or mixtures thereof.

30

It is preferred if the deposition aid is cationic in nature. If a cationic surfactant or cationic softening aid is not present in the formulation it is preferred if a cationic polymeric deposition aid is present. Most preferably the

deposition aid is both cationic in nature and is a fabric softening compound.

5 Mixtures of deposition aids may be used, for example, a mixture of a cationic surfactant and a nonionic surfactant, or a fabric softening compound and a polymeric deposition aid.

10 Suitable cationic deposition aids include water soluble single chain quaternary ammonium compounds such as cetyl trimethyl ammonium chloride, cetyl trimethyl ammonium bromide, or any of those listed in European Patent No. 258 923 (Akzo).

15 However, it is preferred if the deposition aid is a fabric softening compound. In particular substantially water insoluble quaternary ammonium materials comprising a single alkyl or alkenyl chain having an average length equal to or greater than C_{20} are preferred. Even more preferable are
20 compounds comprising a polar head group and two alkyl or alkenyl chains each having an average chain length equal to or greater than C_{14} .

25 Preferably the fabric softening deposition aid of the invention has two long alkyl or alkenyl chains with an average chain length equal to or greater than C_{14} . More preferably each chain has an average chain length greater than C_{16} . Most preferably at least 50% of each long chain alkyl or alkenyl group has a chain length of C_{18} .

30

It is preferred if the long chain alkyl or alkenyl groups of the fabric softening deposition aid are predominantly linear.

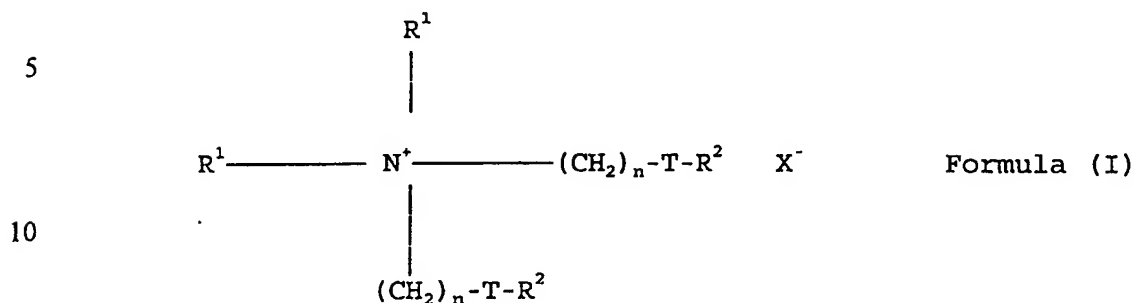
5 The fabric softening deposition aids used in the compositions of the invention are molecules which provide excellent softening, and are characterised by a chain melting $-L\beta$ to $L\alpha$ - transition temperature greater than 25°C , preferably greater than 35°C , most preferably greater than
10 45°C . This $L\beta$ to $L\alpha$ transition can be measured by DSC as defined in "Handbook of Lipid Bilayers, D Marsh, CRC Press, Boca Raton Florida, 1990 (Pages 137 and 337).

"Substantially insoluble" fabric compounds in the context of
15 this invention are defined as fabric compounds having a solubility less than 1×10^{-3} wt% in demineralised water at 20°C . Preferably the fabric softening deposition aids have a solubility less than 1×10^{-4} . Most preferably the fabric softening deposition aids have a solubility at 20°C in
20 demineralised water from 1×10^{-8} to 1×10^{-6} .

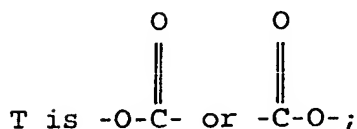
Preferred fabric softening deposition aids are quaternary ammonium compounds, preferably those with at least one ester link.

25 It is especially preferred if the fabric softening deposition aid is a water insoluble quaternary ammonium material which comprises a compound having two C_{12-18} alkyl or alkenyl groups connected to the molecule via at least one
30 ester link. It is more preferred if the quaternary ammonium material has two ester links present. An especially

preferred ester-linked quaternary ammonium material for use in the invention can be represented by the formula (I):



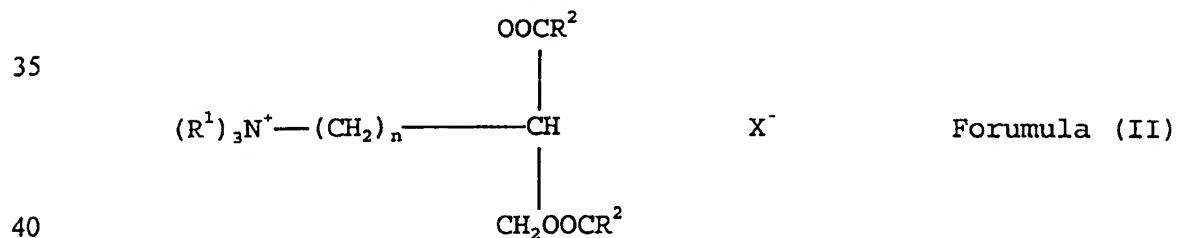
wherein each R^1 group is independently selected from C_{1-4} alkyl, hydroxyalkyl or C_{2-4} alkenyl groups; and wherein each R^2 group is independently selected from C_{8-28} alkyl or alkenyl groups;



X^- is any suitable anion and n is an integer from 0-5.

Di(tallowoxyloxyethyl) dimethyl ammonium chloride is especially preferred.

A second preferred type of quaternary ammonium material can be represented by the formula (II):



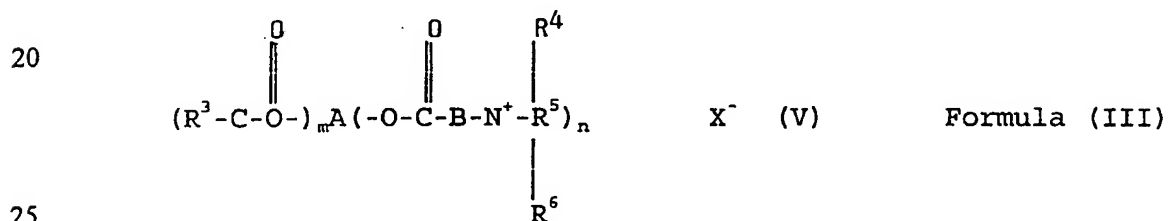
wherein R^1 , n , X^- and R^2 are as defined above.

It is advantageous for environmental reasons if the
 5 quaternary ammonium material is biologically degradable.

Preferred materials of this class such as 1,2 bis[hardened
 tallowoyloxy]-3- trimethylammonium propane chloride and
 their method of preparation are, for example, described in
 10 US 4 137 180 (Lever Brothers). Preferably these materials
 comprise small amounts of the corresponding monoester as
 described in US 4 137 180 for example 1-hardened
 tallowoyloxy -2-hydroxy 3-trimethylammonium propane
 chloride.

15

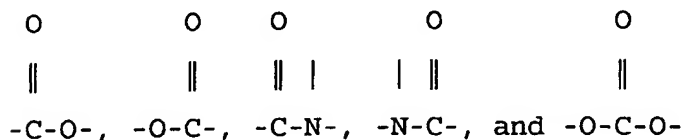
The fabric softening deposition aid of the composition may
 also be compounds having the formula (III):



wherein X is an anion, A is an $(m+n)$ valent radical
 remaining after the removal of $(m+n)$ hydroxy groups from an
 aliphatic polyol having p hydroxy groups and an atomic ratio
 30 of carbon to oxygen in the range of 1.0 to 3.0 and up to 2
 groups per hydroxy group selected from ethylene oxide and
 propylene oxide,
 m is 0 or an integer from 1 to $p-n$, n is an integer from 1
 to $p-m$, and p is an integer of at least 2,

B is an alkylene or alkylidene group containing 1 to 4 carbon atoms,

5 R³, R⁴, R⁵ and R⁶ are, independently from each other, straight or branched chain C₁-C₄₈ alkyl or alkenyl groups, optionally with substitution by one or more functional groups and/or interruption by at most 10 ethylene oxide and/or propylene oxide groups, or by at most two functional groups selected
10 from



15 or R¹¹ and R¹² may form a ring system containing 5 or 6 atoms in the ring, with the proviso that the average compound either has at least one R group having 22-48 carbon atoms, or at least two R groups having 16-20 carbon atoms, or at
20 least three R groups having 10-14 carbon atoms. Preferred compounds of this type are described in EP 638 639 (Akzo).

The deposition aid may also be a nonionic surfactant. Preferred nonionic ethoxylated surfactants have an HLB of
25 from about 10 to about 20. It is advantageous if the surfactant alkyl group contains at least 12 carbon atoms.

Suitable polymeric deposition aids for use with the invention include cationic and nonionic polymeric deposition
30 aids.

Suitable cationic polymeric deposition aids include cationic guar polymers such as Jaguar (ex Rhone Poulenc), cationic cellulose derivatives such as Celquats (ex National Starch), Flocaid (ex National Starch), cationic potato starch such as SoftGel (ex Aralose), cationic polyacrylamides such as PCG (ex Allied Colloids). Cationic polymeric aids are particularly preferred in the absence of any other cationic material in the composition.

Suitable nonionic deposition aids include Pluronic (ex BASF), dialkyl PEGs, cellulose derivatives as described in GB 213 730 (Unilever), hydroxy ethyl cellulose, starch, and hydrophobically modified nonionic polyols such as Acusol 880/882 (ex Rohn & Haas).

Mixtures of any of the aforementioned deposition aids may be used.

Composition pH

The compositions of the invention preferably have a pH from 1.5 to 7, more preferably from 1.5 to 5.

Other Ingredients

The compositions of the present invention can also contain fatty acids, for example $C_8 - C_{24}$ alkyl or alkenyl monocarboxylic acids, or, polymeric carboxylic acids. Preferably saturated fatty acids are used, in particular, hardened tallow $C_{16} - C_{18}$ fatty acids.

The composition can also contain one or more optional ingredients, selected from electrolytes, non-aqueous solvents, pH buffering agents, perfumes, perfume carriers, fluorescers, colorants, hydrotropes, antifoaming agents, antiredeposition agents, polymeric and other thickeners, enzymes, optical brightening agents, opacifiers, anti-shrinking agents, anti-wrinkle agents, anti-spotting agents, germicides, fungicides, anti-oxidants, anti-corrosion agents, drape imparting agents, antistatic agents, sunscreens, colour care agents and ironing aids.

If the product is a liquid it may be advantageous if a viscosity control agent is present. Any viscosity control agent used with rinse conditioners is suitable for use with the present invention, for example biological polymers such as Xanthum gum (Kelco ex Kelsan and Rhodopol ex Rhone-Poulenc), Guar gum (Jaguar ex Rhone-Poulenc), starches and cellulose ethers. Synthetic polymers are useful viscosity control agents such as polyacrylic acid, poly vinyl pyrrolidone, polyethylene, carbomers, cross linked polyacrylamides such as Acosol 880/882 polyethylene and polyethylene glycols.

Also suitable as viscosity modifiers are decoupling polymers and deflocculating polymers.

Product Form

The compositions may be in any physical form conventionally used for fabric softening compositions for example, powder,

paste, gel or liquid. It is preferred if the product is a liquid and especially preferred if it is an emulsion.

5 **Examples**

The invention is illustrated by the following non-limiting examples. Further examples within the scope of the present invention will be obvious to the man skilled in the art.

10

Examples of the invention are illustrated by a number while comparative examples are illustrated by a letter.

15 **A. Preparation of CPE's and RSE's**

Compound 1

Preparation of a glucose penta ester, where 50% (approx) of the ester groups are acetyl groups and 50% (approx) are decanoyl groups.

20 α -D glucose (0.111M) was added to pyridine at room temperature. Acetyl chloride (0.254M) and decanoyl chloride (0.278M) were mixed together and the acid chloride mixture added drop-wise to the glucose solution. The acid chlorides were added slowly over a period of about 1.5 hours. As the reaction proceeded, large amounts of a thick white precipitate were formed and ethyl acetate was added to
30 maintain the fluidity of the reacton mixture.

The reaction mixture was stirred overnight (>16 hours) at room temperature. The reaction mixture was filtered to remove the solid pyridine hydrochloride salt and stripped under vacuum to remove the excess pyridine. The remaining produce was taken back up in ethyl acetate, and combined with water in a separating funnel. The mixture was shaken and allowed to separate.

The organic layer was washed with water, dilute hydrochloric acid (to remove residual pyridine), saturated sodium bicarbonate (to remove the residual carboxylic acids), water (twice) and saturated sodium chloride. The organic layer was dried over magnesium sulphate (anhydrous) overnight, filtered and stripped under high vacuum with heating to leave a pale brown oily residue.

The IR spectrum showed only a single sharp peak at 1749 cm^{-1} in the carbonyl region. This confirms that there is no fatty acid chloride nor fatty acid remaining in the product. In addition, there is no intensity in the $3000\text{-}3500\text{ cm}^{-1}$ region due to un-reacted hydroxyl groups.

An analysis of the ^1H NMR spectrum gives an $\alpha:\beta$ anomer ratio of 34:66. The integrals give a ring proton (3.5-6.5 ppm) to alkyl chain proton (0.8-2.5 ppm) ratio of 0.127. This is agreement with a theoretical ratio of 0.127 for a glucose molecule esterified with an average of 2.5 acetyl groups and 2.5 decanoyl groups per ring.

Hydroxyl value 8.5.

Density 1.0300g/cm³.

Viscosity 810 mPa.s

5

Compound 2

Preparation of a glucose penta ester, where 50% (approx) of the ester groups are acetyl groups and 50% (approx) are
10 dodecanoyl groups.

The same procedure was followed as for Compound 1 with replacement of the decanoyl chloride with dodecanoyl chloride.

15

The IR spectrum showed only a single sharp peak at 1748 cm⁻¹ in the carbonyl region. This confirms that there is no fatty acid chloride nor fatty acid remaining in the product. In addition, there is no intensity in the 3000-3500 cm⁻¹
20 region due to un-reacted hydroxyl groups.

An analysis of the ¹H NMR spectrum gives an α:β anomer ratio of 67:33. The integrals also give a ring proton (3.5-6.5 ppm) to alkyl chain proton (0.8-2.5 ppm) ratio of 0.1039.
25 This is in agreement with theoretical ratio of 0.1076 for a glucose molecule esterified with an average of 2.5 acetyl groups and 2.5 dodecanoyl groups per ring.

Hydroxyl value 19.5
30 Density 1.0078g/cm³
Viscosity 693 mPa.S

Compound 3

Preparation of a glucose penta ester, where 50% (approx) of the ester groups are acetyl groups and 50% (approx) are octanoyl groups.

The same procedure was followed as for Compound 1 with replacement of decanoyl chloride with octanoyl chloride to give a dark brown viscous oil.

The IR spectrum showed only a strong sharp peak at 1748 cm^{-1} and a much smaller peak at 1674 cm^{-1} in the carbonyl region. Again, this confirms that there is no fatty acid chloride nor fatty acid remaining in the product. There is no intensity in the $3000\text{-}3500\text{ cm}^{-1}$ region due to un-reacted hydroxyl groups.

Hydroxyl value undetectable
Density 1.0535 g/cm^3
Viscosity 3500 mPas.S

Compound 4

Preparation of a glucose penta ester, where 70% (approx) of the ester groups are acetyl groups and 30% (approx) are octanoyl groups.

The same procedure was followed as for Compound 1 using acetyl chloride (0.388 m) and octanoyl chloride (0.156m) to give an extremely viscous dark brown oil.

5 The IR spectrum showed only a strong sharp peak at 1749cm^{-1} and a much smaller peak at 1673cm^{-1} in the carbonyl region. Again, this confirms that there is no fatty acid chloride nor fatty acid remaining in the product. There is no intensity in the $3000\text{-}3500\text{cm}^{-1}$ region due to un-reacted
10 hydroxyl groups.

Viscosity 11450 mPa.s

Density 1.1034g/cm³

15

Compound 5

Preparation of sucrose octa ester, where all of the ester groups are oleate groups.

20

Oleic acid (0.40M) (90% technical grade) was mixed with dichloromethane in a large round bottom flask to which Oxalyl chloride (0.44M) in dichloromethane was added. The residual dichloromethane and the excess oxalyl chloride were
25 stripped off under high vacuum to leave 120g of oleoyl chloride.

In a separate flask, sucrose (0.044M) was added to a mixture of pyridine, ethyl acetate and dimethyl formamide. The
30 oleoyl chloride prepared above was added dropwise to the

mixture and the mixture was stirred overnight at 80°C with a small amount of 4-dimethylamino pyridine catalyst.

5 The clear dark brown solution was stripped under vacuum and taken back up in diethyl ether. The organic layer was washed with water (twice), dilute hydrochloric acid, saturated sodium bicarbonate, water (four more times) and saturated brine after which the organic layer was dried over magnesium sulphate overnight. The magnesium sulphate was
10 filtered off and the diethyl ether removed under high vacuum to leave a yellow oil.

The IR spectrum showed a sharp peak at 1740 cm⁻¹ in the carbonyl region. This confirms that there is no fatty acid
15 chloride nor fatty acid remaining in the product.

Hydroxyl value undetectable

Viscosity 568 mPa.s

Density 0.9446 g/cm³

20

Compound 6

Preparation of cellobiose octaester, where all of the ester
25 groups are oleate groups.

A similar procedure was followed as for compound 5 with the replacement of the sucrose by cellobiose. The only minor changes to the procedure were that the reaction solvent
30 comprised a mixture of pyridine and dimethyl formamide only

and the final reaction was heated with activated charcoal prior to drying with magnesium sulphate.

5 The IR spectrum showed a sharp peak at 1744 cm^{-1} in the carbonyl region, confirming that there is no residual fatty acid nor fatty acid chloride remaining in the product.

Gel permeation chromatography shows only one sharp signal. This indicates nearly all the material is the octaester.

10

Hydroxyl value: undetectable

Viscosity: 1040 mPa.s

Density: 0.9459 g/cm^3

15

Compound 7

Preparation of methyl α -D-glucopyranoside tetraester where 37.5% (approx) of the ester groups are acetyl groups and 20 62.5% (approx) are dodecanoyl groups.

A similar procedure was followed as for compound 2 with replacement of the glucose with methyl α -D-glucopyranoside.

25 The IR spectrum showed only a single sharp peak at 1747 cm^{-1} in the carbonyl, region confirming that there is no residual fatty acid, nor, fatty acid chloride remaining in the product.

30

Hydroxyl value: 1.5

Viscosity: 225 mPa.s

Density: 0.9930 g/cm³

5

Compound 8

Preparation of *myo*-inositol hexaester where 50% (approx) of the ester groups are acetyl groups and 50% (approx) are dodecanoyl groups.

10

A similar procedure was followed as for compound 2 with replacement of the glucose with *myo*-inositol.

15 The IR spectrum showed only a single sharp peak at 1755cm⁻¹ in the carbonyl region, confirming that there is no residual fatty acid nor fatty acid chloride remaining in the product.

Hydroxyl value: 0.04

20 Viscosity: 20,500 mPa.s

Density: 1.0227 g/cm³

B. Examples of Compositions

25 In table 1 selected compounds above were placed in an ethanol or acetone solution (0.9g/l) and padded onto fabric to give deposition of 0.2% of compound by weight on the fabric.

Table 1

Example	Softening Active	Solvent
1	Glucose perester 50:50 C ₂ :C ₁₀ Compound 1 (oily liquid)	Ethanol
2	Glucose perester 50:50 C ₂ :C ₁₂ Compound 2 (oily liquid)	Ethanol
3	Glucose perester 50:50 C ₂ :C ₈ Compound 3 (oily liquid)	Ethanol
4	Sucrose octaoleate Compound 5 (oily liquid)	Acetone
5	Sucrose pentaoleate (Ryoto 0-170) (oily liquid)	Acetone
6	Sucrose pentaester (Ryoto POS-135*) (soft solid)	Acetone
7	Sucrose pentataurate (Ryoto L-195) (soft solid)	Acetone
A	Sucrose tetrastearate (Ryoto S-270) (white powder)	Ethyl Acetate

* Contains a mixture of palmitate, stearate and oleate ester groups.

Examples 8 to 21 (Table 2) below were prepared by mixing the listed components together in water. All Examples contain 5% active material.

Table 2: mixtures with CTAC (fabric softening compound)

Example	B	8	9	10	11	12	13	14	15	16	17	18	19	20	21
Cetyl trimethyl ammonium chloride (CTAC)	1	3	2	1	3	2	1	1	1	1	1	1	1	1	1
Glucose perester 50:50 C ₈ :C ₁₂ Compound 2 (oily liquid)		2	3	4											
Glucose perester 50:50 C ₈ :C ₁₀ Compound 1 (oily liquid)					2	3	4								
Sucrose pentaoleate (Ryoto 0-170)								4							
Sucrose pentaester (Ryoto POS-135)									4						
Sucrose pentalaurate (Ryoto L-195)										4					
Sucrose tetrastearate (Ryoto S-270)	4														
Sucrose tetraoleate (oily liquid)											4				
Sucrose pentaerucate (Ryoto ER-190) (soft liquid)												4			
Sucrose tetraerucate (Ryoto ER-290) (soft liquid)													4		
Sorbitan monooleate (Span 80) (oily liquid)														4	
Sorbitan trioleate (Span 85) (oily liquid)															4
Water	95	95	95	95	95	95	95	95	95	95	95	95	95	95	95
CTAC:oil ratio	1:4	3:2	2:3	1:4	3:2	2:3	1:4	1:4	1:4	1:4	1:4	1:4	1:4	1:4	1:4

Span 80 and Span 85 are available from ICI surfactants.
 CTAC is available from Aldrich (25% solution).
 The Ryoto products are available from Mitsibushi-Kagaku Food Corporation.

The examples listed in Table 3 below were prepared by heating the ingredients together at 80°C, and mixing at high shear.

Table 3: mixtures with HEQ (fabric softening compound)

Example	22	23	24	25	26	27	C	28	29	30	31	32	D
HEQ ¹	2.57	1.71	0.86	2.57	1.71	0.86	4.28	0.86	0.86	0.86	2.57	1.71	0.86
Fatty Acid	0.43	0.39	0.14	0.43	0.29	0.14	0.72	0.14	0.14	0.14	0.43	0.39	0.14
Glucose perester 50:50 C ₈ :C ₁₂ Compound 2 (oily liquid)	2	3	4	-	-	-	-	-	-	-	-	-	-
Glucose perester 50:50 C ₈ :C ₁₀ Compound 1 (oily liquid)	-	-	-	2	3	4	-	-	-	-	-	-	-
Sucrose pentaoleate (Ryoto 0-170) (oily liquid)	-	-	-	-	-	-	-	4	-	-	2	3	-
Sucrose pentaester (Ryoto POS-135) (soft liquid)	-	-	-	-	-	-	-	-	4	-	-	-	-
Sucrose pentalaurate (Ryoto L-195) (soft solid)	-	-	-	-	-	-	-	-	-	4	-	-	-
Sucrose tetraesterate (Ryoto S-270) (white powder)	-	-	-	-	-	-	-	-	-	-	-	-	4
Water	95	95	95	95	95	95	95	95	95	95	95	95	95
HEQ:oil ratio	1.285:1	1:1.45	1:4.65	1:13.75	1:1.75	1:4.65	-	1:4	1:4	1:4	3:2	2:3	-

HEQ¹ is 1,2 bis(hardened tallowyloxy)-3- trimethylammonium propane chloride available from Hoechst.

The examples listed in Table 4 prepared by heating the ingredients together at 80°C, and mixing at high shear.

5 Table 4; mixtures with Prapagen 34452

The examples listed in Table 4 were prepared by heating the ingredients together at 80°C and mixing at high shear.

10

Example	33	34	35	36	37	38	E
Prapagen 3445 ²	3	2	1	3	2	1	5
Glucose perester 50:50 C ₂ :C ₁₂ Compound 2 (oily liquid)	2	3	4				-
Glucose perester 50:50 C ₂ :C ₁₀ Compound 1 (oily liquid)	-	-	-	2	3	4	-
Water	95	95	95	95	95	95	95
Prapagen:oil ratio	3:2	2:3	1:4	3:2	2:3	1:4	

² Dimethyl ditallow ammonium chloride.

Table 5; mixtures with Accosoft 460 HC

The examples listed in Tables 5, 6 and 7 were prepared by heating the ingredients together at 80°C and mixing at high shear.

Example	39	40	41	F
Accosoft 460 HC	3	2	1	5
Sucrose pentaoleate Ryoto 0-170 (oily liquid)	2	3	4	0
Water	95	95	95	95
Accosoft:oil	3:2	2:3	1:4	-

10

Table 6; mixtures with Accosoft 550 HC

Example	42	43	44	G
Accosoft 550 HC	3	2	1	5
Sucrose pentaoleate Ryoto 0-170 (oily liquid)	2	3	4	0
Water	95	95	95	95
Accosoft:oil	3:2	2:3	1:4	-

15 Accosoft 460 and Accosoft 550 HC are available from Stepan.

Table 7; mixtures with Arquad HT

Example	45	46	47
Arquad HT	1	1	1
Sucrose Tetraoleate (oily liquid)	4		
Sucrose pentaerueate (Ryoto ER-190) (oily liquid)		4	
Sucrose tetraeureate (Ryoto ER-290) (oily liquid)			4
Water	95	95	95
Arquad HT:oil	1:4	1:4	1:4

5

Softness Evaluation

Softening performance was evaluated by adding 0.1g of fabric softening compound (2ml of a 5% a.d. dispersion for liquids) to 1 litre of tap water, at ambient temperature in a tergotometer. Three pieces of terry towelling (8cm x 8cm, 40g total weight) were added to the tergotometer pot. The cloths were treated for 5 minutes at 65 rpm, spin dried to remove excess liquor and line dried overnight and conditioned at 21°C/65°C/ for 24 hours.

15

For Examples 1 to 7 and A the terry towelling was treated by padding the composition directly onto the fabric as described above.

20

Softening of the fabrics was assessed by an expert panel of 4 people using a round robin paired comparison test protocol. Each panel member assessed four sets of test

cloths. Each set of test cloths contained one cloth of each test system under a evaluation. Panel members were asked to assess softness on a 8 point scale. Softness scores were calculated using an "Analysis of Variance" technique. Lower values are indicative of better softening.

Absorbency Evaluation

The absorbency was measured using the textile and paper industry wicking (Klemm) test. A strip of treated fabric is held vertically with a clip whilst the free end is weighed down with a piece of rubber. The strip is lowered into a tray containing a 0.02% diphenyl red dye solution such that the rubber weight is just below the surface. The height to which the liquid rises up the strip in one hour is measured (unless stated otherwise). Six fabric strips are measured for each treatment. "Active in Table 8 is the deposition aid.

Table 8

Example	Oil	Active	Wicking Height (cm)	Softness Score
1	Compound 1	-	13.50 ²⁰	3.83
2	Compound 2	-	13.52 ²⁰	4.00
3	Compound 3	-	13.12 ²⁰	5.00
4	Compound 5	-	14.83 ²⁰	4.00
5	Ryoto 0-170	-	20.00	2.81
6	Ryoto POS-135	-	10.41	2.25
7	Ryoto L-175	-	8.53	2.75
A	Ryoto S-270	-	4.91	2.37
8	Compound 2	CTAC	14.55	4.17
9	Compound 1	CTAC	15.64	3.83
10	Compound 2	CTAC	16.25	3.67
11	Compound 1	CTAC	15.06	4.83
12	Compound 1	CTAC	16.39	4.58
13	Compound 1	CTAC	17.08	3.83
14	Ryoto 0-170	CTAC	15.88	3.07
15	Ryoto POS-135	CTAC	8.82	3.05
16	Ryoto L-195	CTAC	7.18	3.57
17	Sucrose tetraoleate	CTAC	14.04	4.00
18	Ryoto ER-190	CTAC	15.21	3.90

Example	Oil	Active	Wicking Height (cm)	Softness Score
19	Ryoto ER-290	CTAC	13.35	3.75
20	Span 80	CTAC	13.43	4.56
21	Span 85	CTAC	13.70	4.25
B	Ryoto S-170	CTAC	6.33	3.17
22	Compound 2	HEQ	5.33	2.67
23	Compound 2	HEQ	8.50	2.70
24	Compound 2	HEQ	11.59	3.29
25	Compound 2	HEQ	3.12	3.325
26	Compound 2	HEQ	5.79	3.875
27	Compound 2	HEQ	11.79	4.75
28	Ryoto 0-170	HEQ	11.04	3.08
29	Ryoto POS-135	HEQ	9.41	4.50
30	Ryoto L-195	HEQ	9.95	4.25
31	Ryoto 0-170	HEQ	8.19	2.31
32	Ryoto 0-170	HEQ	9.89	2.65
C	-	HEQ	2.20	2.5
D	Ryoto S-270	HEQ	6.25	3.75
33	Compound 2	Prapagen 3445	11.25	4.12
34	Compound 2	Prapagen 3445	11.83	4.87
35	Compound 2	Prapagen 3445	13.08	4.87
36	Compound 1	Prapagen 3445	13.21	4.37
37	Compound 1	Prapagen 3445	13.75	4.75
38	Compound 1	Prapagen 3445	15.12	4.625

Example	Oil	Active	Wicking Height (cm)	Softness Score
E	-	Prapagen 34452	12.71	5.38
39	Ryoto 0-170	Accosoft 460HC	6.78	3.67
40	Ryoto 0-170	Accosoft 460HC	9.3	3.5
41	Ryoto 0-170	Accosoft 460HC	13.01	3.87
F	Ryoto 0-170	Accosoft 460HC	3.34	4.17
42	Ryoto 0-170	Accosoft 550HC	10.97	3.92
43	Ryoto 0-170	Accosoft 550HC	12.76	3.8
44	Ryoto 0-170	Accosoft 550HC	15.23	4.17
G	Ryoto 0-170	Accosoft 550HC	9.9	5.15
45	Sucrose tetraoleate	Arquad HT	14.00	3.94
46	Ryoto ER-190	Arquad HT	14.73	4.25
47	Ryoto ER-290	Arquad HT	13.58	4.00
Desized towelling			20.00	8.0

These wicking heights were measured after only 30 minutes.

- 5 The above softness and absorbency results demonstrate that the examined compounds except excellent hydrophilic softening properties when compared to the compounds of the prior art.

Examples 48 and 49

Examples 48 and 49 were prepared as 5% total active emulsions/dispersion in water of a 1:4 fabric softener:sucrose ester mixture. 0.3% of a perfume was included in the compositions which were then tested for perfume longevity. See Table 11 below.

10 Table 11

Example	Fabric Softener	Sucrose ester	Wet	Perfume result	
				5 hours	24 hours
48	CTAC	Sucrose penta-oleate (Ryoto 0-170) liquid	accept-able	accept-able	accept-able
49	HEQ	Sucrose penta-steate as above	3.0	2.4	1.76
H	HEQ	-	3.1	1.9	1.19

Perfume intensity was measured by a trained panel except for example 48 which was measured separately. A value of 0.5 is given for a cloth immersion in fabric. Five represents a very strong smell and 0 an undetectable smell. The perfume intensity is assessed after application (when the cloth is wet), after 5 hours and after 24 hours.

20 It can be seen that the presence of the oil increases perfume longevity.

Example 50

Example 50 was prepared as a series of a 5% total active emulsions/dispersions in water of 4.5% sucrose tetraerucate (oily liquid, Ryoto ER 290), 0.5% CTAC and 0.2% of a polymer deposition aid (some of which are cationic) as given below:-

Example 50a	Celquat SC240	(ex National Starch & Chemical)
Example 50b	Coltide HQS	(ex Croda Colloids)
10 Example 50c	Crodacel QS	(ex Croda Colloids)
Example 50d	FlocAid 34	(ex National Starch)
Example 50e	Bermacoll CST 035	(ex Berol Nobel) Nonionic
Example 50f	Merquat 100	(ex Chemviron)
Example 50g	Softgel BDA	(ex Avebe)

15

Example 51

Example 51 was prepared as a series of 5% total active emulsions/dispersions in water of 4.5% sucrose tetraerucate (as in Example 55), 0.5% of a nonionic surfactant deposition aid (Synperonic A7) and 0.2% of the following deflocculating polymers (all of which are cationic).

Example 51a	Celquat SC240	(ex National Starch & Chemical)
25 Example 51b	Coltide HQS	(ex Croda Colloids)
Example 51c	Coltide QS	(ex Croda Colloids)
Example 51d	Poly DMDAAC:EHA	(ex National Starch) - 10:1 molar ratio copolymer (Mwt 2400 approx)

Example 52

Example 52 was prepared as a 1:4 emulsion/dispersion (5%
total active) of DEEDMAC:sucrose pentaoleate (Ryoto 0-170)
5 by mixing at high temperatures.

The softening and absorbency was measured as before. The
composition was stable and the results are given below:-

10 Softening 4.00
Wicking height 11.74 cm

Example 53

15 A fully formulated fabric softening composition as according
to the present invention was prepared as below:

	<u>% by weight</u>
Cetyl trimethyl ammonium chloride	1.0
20 Sucrose ester oil	4.0
Cationic polymer deposition aid	0.2
Dye	0.0025
Perfume	0.3
Minors	0.02
25 Water	balance

The sucrose ester oil was Ryoto ER290 available from
Mitsubishi Kagaku Foods Corporation. The deposition aid was
Floc Aid 34 available from National Starch & Chemical.

30

Example 54

Table 9 below shows the T₂ NMR solid:liquid ratio of CPE's and RSE's used according to the present invention. A comparative crystalline solid sugar ester is also included. The ratios were measured at 20°C. The degree of esterification letherification is stated.

10 Table 9

Material	Solid:Liquid ratio at 20°C	Degree and % of esterification		Physical Form
Ryoto S270 (comparative)	96:4	4/8	50%	crystalline solid. Liquid
Ryoto 0-170	0:100	5/8	62.5%	liquid
Ryoto ER-290	0:100	4/8	50%	liquid
Ryoto ER-190	0:100	5/8	50%	soft solid
Ryoto POS-135	30:70	5/8	62.5%	soft solid
Ryoto L-195	43:57	5/8	62.5	liquid
Sucrose tetraoleate	0:100	4/8	50%	liquid
Compound 1 oleate	0:100	5/5	100%	liquid
Compound 2	0:100	5/5	100%	liquid
Compound 3	0:100	5/5	100%	liquid
Compound 4	0:100	5/5	100%	liquid
Compound 5	0:100	8/8	100%	liquid
Compound 6	0:100	8/8	100%	liquid
Compound 7	0:100	4/4	100%	liquid
Compound 8	0:100	6/6	100%	liquid
Span 80	0:100	1/4	25%	liquid
Span 85	0:100	3/4	75%	liquid

The Ryoto materials have previously been described.

Compounds 1 to 8 are described herein above. Span 80 and 85

are Sorbitan monoleate and trioleate oily liquids
respectively, available from ICI surfactants.

Claims

- 1) A fabric softening composition comprising:
 - 5 i) a liquid or soft solid derivative of a cyclic polyol (CPE) or of a reduced saccharide (RSE) resulting from 35 to 100% of the hydroxyl groups in the cyclic polyol or reduced saccharide being esterified or etherified, the
10 derivate (CPE or RSE) having at least 2 or more of ester or ether groups independently attached to a C₈-C₂₂ alkyl or alkenyl chain or mixtures thereof, and containing at least 35% tri or higher esters.;
 - 15 ii) a deposition aid.
- 20 2) A fabric softening composition according to claim 1 wherein the CPE or RSE results from a cyclic polyol or a reduced saccharide having 40-70% of the hydroxyl groups esterified and/or etherified.
- 25 3) A fabric softening composition according to either of claims 1 or 2 wherein the CPE or RSE results from a cyclic polyol or reduced saccharide having 4 or more hydroxyl groups etherified or esterified.
- 4) A fabric softening composition according to any
preceeding claim in which CPE or RSE is derived from a monosaccharide or disaccharide.
- 30 5) A fabric softening composition according to any one of the preceeding claims in which the CPE is a saccharide

polyester, a saccharide polyether or a saccharide polyester/polyether.

- 5 6) A fabric softening composition according to any preceeding claim wherein the CPE is a selected from sucrose pentalaurate, sucrose tetraoleate, sucrose pentaerucate and sucrose tetraerucate.
- 10 7) A fabric softening composition according to any preceding claim in which the ester or ether groups of the CPE or RSE are independently of one another attached to a C₈ to C₂₂ alkyl or alkenyl chain or a C₂ to C₈ chain and in which the ratio of C₈ to C₂₂ groups to C₂-C₈ is from 5:3 to 3:5.
- 15 8) A fabric softening composition according to any preceding claim in which the deposition aid is selected from a cationic surfactant, a nonionic surfactant, an anionic surfactant, a polymeric deposition aid, a fabric softening compound or mixtures thereof.
- 20 9) A fabric softening composition according to any preceding claim in which the fabric softening compound has a solubility of less than 1×10^{-3} wt% in demineralised water at 20°C.
- 25 10) A fabric softening composition according to claim 9 in which the fabric softening compound is a quaternary ammonium compound.

- 11) A fabric softening composition according to any preceding claim in which the ratio of CPE or RSE to deposition aid is within the range from 1:10 to 15:1.
- 5 12) A fabric softening composition according to any preceding claim which is in liquid form or emulsion form.
- 10 13) A fabric softening composition according to claim which further comprises a viscosity control agent.
- 15 14) Use of a CPE or RSE as defined in any of the preceding claims within a fabric softening composition as a fabric softening aid that does not decrease the fabrics absorbency.
- 20 15) A liquid or soft solid fatty acid ester of glucose having a solid:liquid ratio of between 50:50 and 0:100 as measured by T_2 relaxation time NMR at 20°C, the ester groups comprising a mixture of C_2 - C_{22} alkyl or alkenyl chains and resulting from a cyclic polyol having 35-100% of the hydroxyl groups esterified.
- 25 16) A fatty acid ester of glucose according to claim 15, the ester groups consisting essentially of a C_2 - C_8 alkyl chain and a C_8 to C_{12} straight alkyl chain, the molar ratio of C_2 alkyl chain to C_8 to C_{12} straight alkyl chain being from 2:1 to 1:2.
- 30 17) Use of a sorbitan mono, di or trioleate as a hydrophilic fabric softener.

INTERNATIONAL SEARCH REPORT

In International Application No

PCT/EP 97/05762

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C07H13/02 C11D3/00 C11D1/66 C07H13/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C07H C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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"&" document member of the same patent family

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INTERNATIONAL SEARCH REPORT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>US 4 800 038 A (BROZE GUY ET AL) 24 January 1989 cited in the application see column 1, line 63 - line 64 see column 8, line 17 - line 60 see claims 1,5; example -----</p>	<p>1,7,15, 16</p>

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